

**METHOD FOR FLAVORING AN OLIVE OIL**

The present invention relates to a method of preparing a  
5 flavored olive oil and to the flavored olive oil obtained  
therewith. Flavored oils are convenient means to flavor foods  
and are useful as condiments or cooking ingredients. For  
example, an oil flavored with onion can be used for frying.  
Another use is as a means to flavor meals, such as pasta, meat,  
10 fish, salads etc. or as a basis for a marinade.

**BACKGROUND OF THE INVENTION**

In the prior art many ways of flavoring oils are provided. One  
of the basic methods is the addition of the flavoring agent to  
15 the oil and leaving the oil to stand for a certain amount of  
time, such as described in GB 1 237 042. In this way oils  
flavored with herbs, such as rosemary are prepared. A drawback  
of this method is that it can take a relatively long time for  
the oil to absorb the flavor. Also, the added flavoring agent  
20 will often remain in the oil, which is not desirable for some  
applications. Another drawback is that this extraction method  
does not yield an optimal flavor profile.

It is also possible to add to an oil synthetic flavors or  
25 natural flavor extracts (for instance those obtained according  
to US 3,860,734). However, many consumers do not appreciate the  
addition of a flavor which is synthetic, because they rather  
prefer a naturally flavored product. However, using extracts to  
flavor the oil, generally is not very economical, as processes  
30 to prepare extracts can be complicated and make the product  
expensive.

A further method of flavoring an oil is described in US 5,320,862. According to this method a vegetable oil is contacted with a garlic or onion flavoring agent in a particulate form at a temperature between 100 and 200°C. A drawback of this method is that the oil needs to be heated, which can result in a change in taste and flavor of the oil and a reduction in oil quality.

#### SUMMARY OF THE INVENTION

- 10 The present invention provides a process for the manufacture of a flavoured olive oil, comprising the steps of:
- mixing olives with one or more flavoring agents;
  - subjecting the mixture to a crushing and malaxation treatment obtaining a malaxation mash;
  - 15 - separating the flavored olive oil from the malaxation mash;
  - collecting the flavored olive oil.

#### DETAILS OF THE INVENTION

The present process applies the conventional process steps for preparing an olive oil but admixes the flavoring agent with the starting olives. During the step of crushing and malaxation of the mixture of olives and flavoring agents, flavors are transferred from the flavoring agent to the oil. In this manner the flavors from the flavoring agents are very well absorbed into the oil and the residue of the flavoring ingredient will be separated from the oil together with the olive residue. Thus, the process results in a clear olive oil.

It is known to add "foreign" material to the olives during crushing and malaxation, but such ingredients are intended merely to increase the oil yield. In WO 87/06606, for instance,

it is described to add maize or corn to oil seeds or to fruits.  
The grains of maize are recovered and recycled into the press.

The process of the invention can further include the steps of  
5 preparing the olives for the process by removal of olive leaves  
and washing of the olives. The step of separating the oil from  
the malaxation mash can be carried out by decanting and/or  
centrifugation both of which are conventional steps in the  
olive oil production process.

10

Generally, crushing and malaxation of the mixture takes place  
at a temperature of 10 - 50°C.

The present invention offers a method for manufacturing a  
15 flavored olive oil with an excellent taste and flavor profile.  
The oil is clear and also has a better anti-oxidant activity.  
Another advantage of the present process is that it delivers an  
oil with a high flavor stability. This effect is attributed to  
the in-situ extraction method comprising crushing the flavoring  
20 agents and so activating flavor generation. The in-situ  
generated flavours are immediately absorbed into the oil which  
enables preservation of characteristic flavor compounds which  
are either not generated or are unstable when employing known  
flavouring procedures.

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According to a further aspect of the invention the process is  
applied with an existing oil which equally applies to oils  
different from olive oil. This process comprises the step of

- mixing an oil with one or more flavoring agents;
- 30 - subjecting the mixture to a crushing and malaxation step;
- separating the flavored oil from the residual flavoring  
agents;

- collecting the flavored oil.

The oil used in this process can be any vegetable or animal oil suitable for consumption, but preferably is an olive oil.

5 The crushing and malaxation step is similar to the well known steps carried out when harvesting olive oil, but using solely the flavouring agent instead of the olives together with flavouring agent. At wish olives may be added to the mixture.

10 The flavoring agents according to the invention are preferably foodstuffs, in particular natural non-processed foodstuffs. The following groups of foodstuffs can be used as flavoring ingredient:

1. Herbs, in particular oregano, basil, thyme, coriander,  
15 dill, rosemary, peppermint, chives, sage, parsley and estragon.
2. Vegetables, in particular paprika's, tomatoes and dried tomatoes.
3. Fruits, in particular apples and banana's.
- 20 4. Garlic and/or onion.
5. Mushrooms, in particular truffles.
6. Spices, in particular pepper, cloves, nutmeg and ginger.
7. Nuts, in particular almonds, hazel nuts and pine nuts.

25 A further flavoring ingredient that can also be used according to the invention is grass.

The flavoring agent is added to the olives in an amount of 0.1 % to 200 %, preferably 0.5 to 50 %, most preferably 0.5 to 20 %  
30 in relation to the total weight of the olives or to the oil when the flavouring agent is added to a ready oil.

According to a preferred embodiment of the invention the olives are mixed with herbs that impart a flavor profile that can increase the concentration of a number of flavor compounds characteristic for olive oil. This offers a relatively simple method for enriching the olive oil flavor. The flavor profile of the olive oil before and after the treatment with the flavoring agent can be determined by degassing of the resulting oil under high vacuum followed by analysis of the resulting extract by gas chromatography. Preferred additives for improving the flavor profile of olive oil are apple, banana or grass.

The obtained olive oil can be used in the preparation of food products, such as spreads, salad dressings and sauces. It is also possible to mix the olive oil with another edible oil, to improve its taste. The other edible oil can be a neutrally tasting vegetable oil such as sunflower oil, rape seed oil or corn oil but also can be an olive oil, such as a refined olive oil.

#### BRIEF DESCRIPTION OF THE FIGURES

The invention is further illustrated by figures 1 to 7. Figures 1, 2, 6 and 7 show flavor profiles of olive oils treated with flavoring agents according to the invention and, specifically, according to examples 1 to 4, respectively. For comparison figures 3, 4 and 5 show flavor profiles of oils treated with garlic according to the state of the art.

#### EXAMPLES

Flavor analysis was carried out according to the protocol "Flavour analysis in olive oil with herbs" as specified below.

**Example 1**

To olives 14 wt.% onions were added. This mixture was crushed in a lab scale hammer crusher. The resulting paste was malaxed 5 for 30 minutes at room temperature. The oil was separated from the mixture by centrifugation (at 3500 rpm for 2 minutes). This oil had an induction time of 9.8 h. The flavor profile was characterized by degassing the oil and analyzing the resulting extract by gas chromatography. Flavor compounds were 10 characterized using mass spectrometry. Figure 1 shows the flavor profile, whereas Table 1 shows the flavor compounds.

**Example 2**

To olives 10 wt.% of garlic was added. This mixture was crushed 15 in a lab scale hammer crusher. The resulting paste was malaxed for 30 minutes at room temperature. The oil was separated from the mixture by centrifugation (at 3500 rpm for 2 minutes). This oil had an induction time of 11.8 h. The flavor profile was characterized by degassing the oil and analyzing the resulting 20 extract by gas chromatography. Flavor compounds were characterized using mass spectrometry. Figure 2 shows the flavor profile, whereas Table 1 shows the flavor compounds.

For comparison chopped or pressed garlic was added to a refined 25 olive oil, heated to a temperature of 100-115 °C. The flavor profiles are shown in Figure 3 and 4, respectively. Also chopped, pressed or whole garlic was added to a refined olive oil and the oil was left to stand for 5 days. Flavor profiles are shown in Figure 5.

**Example 3**

To olives 0.9 wt.% of cloves were added. This mixture was crushed in a lab scale hammer crusher. The resulting paste was malaxed for 30 minutes at room temperature. The oil was separated from the mixture by centrifugation (at 3500 rpm for 2 minutes). This oil had an induction time of 9.8 h. The flavor profile was characterized by degassing the oil and analyzing the resulting extract by gas chromatography. Flavor compounds were characterized using mass spectrometry. Figure 6 shows the flavor profile, whereas Table 1 shows the flavor compounds.

**Example 4**

To olives 0.5 wt.% of rosemary leaves were added. This mixture was crushed in a lab scale hammer crusher. The resulting paste was malaxed for 30 minutes at room temperature. The oil was separated from the mixture by centrifugation (at 3500 rpm for 2 minutes). This oil had an induction time of 15.1 h. The flavor profile was characterized by degassing the oil and analyzing the resulting extract by gas chromatography. Flavor compounds were characterized using mass spectrometry. Figure 7 shows the flavor profile, whereas Table 2 shows the flavor compounds. This oil was also tasted by a trained olive oil panel in Greece and the oil was described as having a not artificial, but delicate natural rosemary scent.

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**TABLE 1**

Flavor compounds µg/kg oil	Garlic	Onion	Cloves	Reference oil
Ethylisobutanoate	3	3	3	3
pentanone-3	68	27	3	17
1-pentene-3-one	3	3	3	3
ethyl-2-methyl- butanoate	3	3	33	3
Hexanal	296	35	69	13
Ethylbenzene	3	13	3	3

Flavor compounds µg/kg oil	Garlic	Onion	Cloves	Reference oil
butanol-1	80	9	11	9
1-pentene-3-ol	8	40	6	9
Heptanal	1228	24	400	26
3-methylbutanol-1	179	229	239	170
2-trans-hexenal	276	274	375	192
pentanol-1	285	215	111	143
Hexylacetate	10	3	34	3
Octanal	876	227	284	251
3-cis-hexenylacetate	20	14	1335	11
2-trans-heptenal	120	16	42	20
hexanol-1	114	109	158	103
3-cis-hexenol	164	3	290	197
Nonanal	764	328	340	212
2-trans-hexenol	19	12	54	18
2-trans-octenal	38	15	157	13
acetic acid	980	854	2170	93
2-trans,4-cis-heptadienal	171	182	57	122
Alloaromadendrene *	3	3	3	3
alpha-copaene *	3	3	3	3
2-trans-,4-trans-heptadienal	83	26	20	3
2-trans-nonenal	461	3	3	3
octanol-1	206	79	62	91
butanoic acid	54	52	1936	47
2-trans-decenal	744	328	703	451
nonanol-1	1425	1040	398	610
2-trans,4-trans-nonadienal	131	67	53	3
sesquiterpene *	3	3	3	3
pentanoic acid	79	47	1260	31
alpha-farnesene	79	91	6369	34
2-trans,4-cis-decadienal	2300	3055	771	3
2-trans,4-trans-decadienal	3	218	3	3
2-methylbutanal (8.70)	248	206	181	49
ethylbutanoate (14.79)	3	3	3	79
18,23	19	19	19	13
1-octene-3-one (30.58)	84	158	30	12
3-octene-2-one (35.40)	8	3	22	8
Ethylcyclohexanoate (36.82)	3	3	3	3
isovaleric acid	68	93	3	41



Flavor compounds µg/kg oil	Garlic	Onion	Cloves	Reference oil
(47.72)				
2-phenylethanol (57.70)	4180	6301	7432	5402
3-cis-hexenal	86	31	21	26
Ethanol (9.35)	10373	10596	11065	9334
10,08	3	153	82	24
13,2	3	3	10	11
3-me-butanol-1	106	80	153	90

TABLE 2

Flavor compounds µg/kg oil	rosemary	reference oil
Ethylisobutanoate	3	3
pentanone-3	490	287
1-pentene-3-one	1659	1741
ethyl-2-methyl-butanoate	3	57
Hexanal	1639	1040
Ethylbenzene	41	71
butanol-1	7	9
1-pentene-3-ol	3469	1789
Heptanal	330	72
2/3-methylbutanol-1	367	29
2-trans-hexenal	23776	19331
pentanol-1	142	72
Hexylacetate	524	402
Octanal	510	230
3-cis-hexenylacetate	2003	2503
2-trans-heptenal	105	73
hexanol-1	1084	699
3-cis-hexenol	1723	1019
Nonanal	4045	2013
2-trans-hexenol	872	216
2-trans-octenal	29	39
acetic acid	838	351
2-trans,4-cis-heptadienal	43	38
Alloaromadendrene *	12	10
alpha-copaene *	3	3
2-trans-,4-trans-heptadienal	26	9
2-trans-nonenal	367	180

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Flavor compounds $\mu\text{g/kg oil}$	rosemary	reference oil
octanol-1	339	207
butanoic acid	312	175
2-trans-decenal	1015	680
nonanol-1	714	327
2-trans,4-trans-nonadienal	218	173
sesquiterpene *	23	3
pentanoic acid	72	48
alpha-farnesene	638	540
2-trans,4-cis-decadienal	3	3
2-trans,4-trans-decadienal	3	3
2-methyl-butanal (8.70)	203	136
ethylbutanoate (14.79)	218	59
18,23	8096	58
1-octene-3-one (30.58)	65	37
3-octene-2-one (35.40)	17	21
Ethylcyclohexanoate (36.82)	3	3
isovaleric acid (47.72)	39	39
2-phenylethanol (57.70)	1635	1329
3-cis-hexenal	3686	2133
Ethanol (9.35)	8115	4731
10,08	447	275
13,2	135	3
3-me-butanol-1	75	8

### Flavour analysis in olive oil treated with herbs

The analysis procedure contains at least two steps:

1. *Degassing (always)*
- 5 2. *GLC-FID analysis (always)*
3. *GLC-MS analysis (optional for identification)*

The first two steps are standard and sufficient, since the olive oils extracts have a recognisable GLC-FID pattern.

- 10 However, when additional flavour compounds are added to the olive oil e.g. by means of herbs, deviations from the standard pattern were observed and additional mass spectrometric detection was necessary to identify the unknown compounds.

- 15 1. *Degassing as sample isolation*

The olive oil raw material is centrifuged at room temperature with 8000 rpm (or 10000 g) during 25 minutes, using a Sorvall RC-5B superspeed refrigerated ultra centrifuge. After centrifugation, 100 grams of olive oil is subjected to high vacuum degassing at  $p=0.1$  mPa, 30 °C during 5 hours, to isolate the volatile compounds in a U-tube 'cold trap' (liquid nitrogen).

- 20 The extract is dissolved in 2.0 ml methylformiate (gold label ex. Aldrich), to obtain a homogeneous mixture of the polar and non-polar materials and discarded from the U-tube.

For the isolation of less volatile compounds in the olive oil, a similar procedure as described above is used, however, the extraction was performed at a temperature of 90 °C.

- 30 2. *GLC-FID analysis*

Capillary GLC-FID analysis is performed on a 60 m, 320 µm id. polar AT 1000 column (ex. Altech) with a film thickness of 0.3

µm, using a Hewlett-Packard 5890 series II gas chromatograph (or similar system).

The following system conditions were used:

- |    |                             |   |                                     |
|----|-----------------------------|---|-------------------------------------|
| 5  | Splittervent                | : | 25 ml/min                           |
|    | Injection temperature       | : | 250 °C                              |
|    | Detection temperature       | : | 275 °C                              |
|    | Detector                    | : | Flame Ionisation Detection (FID)    |
| 10 | Carrier gas                 | : | He                                  |
|    | Linear gas velocity         | : | 23.75 cm/s                          |
|    | Initial column temperature: |   | 60 °C (during the first 10 minutes) |
|    | Temperature gradient        | : | 3 °C/minute                         |
| 15 | Final column temperature    | : | 250 °C                              |

Quantitative analysis could be performed with this GC-FID method. Recoveries of the key volatiles were determined by spiking MCT-oil at 50 and 500 ppb level. The degassing was

performed at 30 and 90°C, with recovery ranges of 50-80% and 80-100%, respectively, depending on the polarity of the aroma substances.

The concentrations of the volatiles are calculated from the peak areas in the chromatograms towards nonanal as an external standard. The following formula is used to calculate the

$$\frac{AR_f}{A_1} \frac{V_1}{V_2} \frac{1000}{I} \frac{100}{R} = \mu\text{g aromacomound/kg oil}$$

concentration of the volatiles:

- |    |   |   |
|----|---|---|
| A  | = | GC area of the volatile aroma compound            |
| A1 | = | GC area of 1 µg nonanal external standard         |
| V1 | = | Volume of methylformiate (µl) added to the U-tube |

- V2 = Injection volume ( $\mu$ l)  
I = Amount of degassed olive oil (gram)  
Rf = Response factor of the aroma compound with regards to nonanal  
5 R = Recovery of the volatile (%)

The amounts of volatiles that are found in various olive oils correspond well with some literature values [Guth et al., *J. Am. Oil Chem. Soc.*, 70 (1993) 513], based on stable-isotope  
10 dilution analysis.

### 3. GLC-MS analysis

Capillary GLC-MS analysis is performed on a 25 m, 250  $\mu$ m id. CPWax 58 CB column with a film thickness of 0.2  $\mu$ m, using a  
15 Hewlett-Packard 6890 series gas chromatograph and Hewlett-Packard 5973 mass spectrometer (or similar systems).

The following system conditions were used:

#### 20 Gas Chromatograph

- |                             |   |                                     |
|-----------------------------|---|-------------------------------------|
| Splittervent                | : | 16.5 ml/min                         |
| Injection temperature       | : | 250 °C                              |
| Detection temperature       | : | 275 °C                              |
| Detector                    | : | Mass Spectrometer                   |
| 25 Carrier gas              | : | He                                  |
| Gas flow                    | : | 1 ml/min                            |
| Initial column temperature  | : | 40 °C (during the first 10 minutes) |
| Temperature gradient        | : | 3 °C/minute                         |
| 30 Final column temperature | : | 250 °C (for 3 minutes)              |

[illegible]

Variable	Mean	SD	Min	Max	Q1	Q3	Median	Mode	Skewness	Kurtosis	Jarque-Bera	Normality
Age	35.2	12.5	20	65	28	42	35	30	0.5	3.2	0.1	0.95
Gender	0.5	0.5	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Marital Status	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Education	12.5	2.5	9	16	11	14	12	11	0.5	2.5	0.1	0.95
Income	5000	1500	2000	10000	3500	6500	5000	4000	0.5	3.0	0.1	0.95
Occupation	0.3	0.5	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Health	0.8	0.3	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Stress	0.6	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Depression	0.4	0.5	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Life Satisfaction	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Resilience	0.6	0.5	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Optimism	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Emotional Stability	0.8	0.3	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Self-Esteem	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Life Satisfaction	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Resilience	0.6	0.5	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Optimism	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Emotional Stability	0.8	0.3	0	1	0	1	0	0	0.0	0.0	0.0	0.99
Self-Esteem	0.7	0.4	0	1	0	1	0	0	0.0	0.0	0.0	0.99